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Morphology of LDPE/PA-6 blends compatibilised with poly(ethylene-graft-ethylene oxide)s

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Abstract

In the present study, we have investigated the morphology and thermal properties of blends of low density polyethylene (PE) and polyamide-6 (PA-6) with two poly(ethylene-graft-ethylene oxide)s (PE-PEO) as compatibilisers. Blends with PE/PA-6 ratios between 70:30 and 30:70 and copolymer contents between 0 and 6% were prepared by melt mixing in a Brabender Plastograph. The morphology of the blends was studied by SEM. Addition of PE-PEO2000 to the PE/PA-6 blends decreased the size of the dispersed phase domains significantly and increased the adhesion between the phases. The addition of PE-PEO750 did not have any measurable effect on the PE/PA-6 blend. At a PE/PA-6 ratio of 40:60, the blend formed a morphology with two continuous phases. Addition of 4% PE-PEO2000 to this blend improved the adhesion between the phases significantly. © 2001 Published by Elsevier Science Ltd.

Keywords: Polymer blend; Morphology; Polyethylene

1. Introduction

The demands for polymeric materials with a unique combination of properties are ever growing. A cost efficient solution to meet the demands is the use of polymer blends. By blending different polymers, new materials with a desirable combination of properties can be developed [1]. Most polymers are incompatible and on mixing, they generally form multiphase morphologies. The structures obtained are usually unstable, and the mechanical properties of the blends are poor because of lack of adhesion between the phases. However, the morphology and the properties of the blend can be controlled by introduction of a third component, a compatibiliser, to the blend. The compatibiliser is usually a block or graft copolymer [2–4], which is interfacially active and adsorbs at the interface between the matrix and the dispersed phase, thus controlling and stabilising the blend morphology, and increasing the adhesion between the phases.

The reasons for blending polyolefins and polyamides are either to improve particular properties of the polyamides, such as toughness, moisture absorption or processability, or to improve the performance of the polyolefins with respect to properties such as rigidity, thermal stability and barrier properties to oxygen and solvents. Multilayered materials obtained from PE and PA-6 have been widely used for packaging where PA-6 is incorporated as a barrier layer in PE [5].

Polyethylene (PE) and polyamide-6 (PA-6) are incompatible and will form a phase separated unstable morphology when blended. To stabilise the PE/PA-6 blends, many different compatibilisers have been used [5–10], for example, copolymers or adducts of maleic anhydride [6,8], and acrylate copolymers such as poly(ethylene-graft-butylacrylate) [9] and ethylene/methacrylic acid/isobutyl acrylate terpolymer [10]. We have recently reported on blends of PE and PA-6, in which poly(ethylene-graft-ethylene oxide) and poly(ethylene-*co*-acrylic acid) were used as compatibilisers [11].

In the present work, we report an extended study of compatibilisation of low density PE and PA-6 blends using two poly(ethylene-graft-ethylene oxide)s (PE-PEO) as compatibilisers. The objective was to investigate the effect on blend morphology of the addition of graft copolymer to PE/PA-6 blends with varying compositions.

2. Experimental

2.1. Materials

Low density PE LD SC 7641 (Dow, density 0.9235 g/cm^3, melt index 2.0 g/min (190°C/2.16 kg)) and Ultramid B4 PA-6

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Table 1
Compositions and thermal properties of the graft copolymers

Copolymer	M_n grafts (g/mol)	MPEO content[a] (%)	T_m(MPEO)[b] (°C)	T_m(PE)[b] (°C)
PE-PEO2000	2000	37	41	92
PE-PEO750	750	40	8	93

[a] Measured by NMR.
[b] Measured by DSC.

(BASF, MFR 13 (230°C/5 kg)) were used as blend components. The graft copolymers, PE-PEO, containing poly(ethylene oxide) side chains on a PE backbone was prepared by coupling of poly(ethylene-*co*-acrylic acid) and poly(ethylene oxide) monomethyl ether (MPEO) by esterification. PEAA and MPEO were allowed to react in *o*-xylene at 140°C under N_2 atmosphere in the presence of *p*-toluenesulfonic acid as catalyst. After the reaction was finished, butanol was added in excess to esterify residual acid groups. Details of the synthesis are given elsewhere [12]. Data on composition and thermal transitions for the copolymers are listed in Table 1.

2.2. Blend preparation

Blends of PE and PA-6 containing 0–6 wt% graft copolymer were prepared in a Brabender Plastograph using a W50E mixing chamber. PE and PA-6 were dried under vacuum at 80°C for 24 h. The graft copolymers were dried at ambient conditions for 48 h, and under vacuum for 24 h. A dry-blended mixture of the polymers, having a total weight of 38 g was fed to the mixer head. The mixing was allowed to proceed for 5 min at a temperature of 240°C in an inert atmosphere of N_2, and at a roller speed of 30 rpm. The blends were discharged from the mixer and cooled at ambient conditions. The compositions of the blends are given in Table 2.

2.3. Sample preparation for morphology analysis

Scanning electron microscopy (SEM) was used to study the morphology of the blends. To prepare samples for SEM analysis, the blends were compression moulded into plates approximately 0.4 mm thick. The moulding temperature was 240°C for all blends. The 70:30 blends were also moulded at 170°C. From the plates, 10 mm wide test bars were cut. The bars were quenched in liquid N_2 and freeze fractured. The fractured sample surfaces were sputter coated with a 15 nm thick layer of Au/Pd. All SEM studies were performed using an ISI 100A instrument operating at 15 kV.

Table 2
Blend compositions

Additive	PE/PA ratio	Copolymer concentration (%)
PE-PEO2000	70/30	0, 0.5, 1, 2, 4 and 6
	60/40	0 and 4
	50/50	0 and 4
	40/60	0 and 4
	30/70	0, 1, 2 and 4
PE-PEO750	70/30	0 and 4

2.4. Evaluation of the dispersed phase size

In order to measure the size and size distribution of the PA-6 domains of the virgin blends from the mixer, the PE phase was dissolved and separated from the PA-6 phase. Pieces of the blends were immersed in *o*-xylene at 140°C for 3 h, leaving the PA-6 domains as a suspension of particles. The suspension was allowed to settle for 3.5 h, and then, the top phase containing PE was removed and the bottom layer containing the PA-6 domains was redispersed in *o*-xylene. This procedure was repeated three times for all samples. The remaining PA-6 phase was then transferred to SEM holders and sputter coated as above. The number average diameter, $\bar{D}_n$, and the weight average diameter, $\bar{D}_w$, of the PA-6 domains were evaluated from micrographs of the isolated PA-6 domains. Approximately 2000 PA-6 particles were measured for each sample. The average diameters are defined as

$$\bar{D}_n = \frac{\sum n_i D_i}{\sum n_i}$$

$$\bar{D}_w = \frac{\sum n_i D_i^4}{\sum n_i D_i^3}$$

In the 30:70 blends, with PA-6 being the matrix, the freeze-fractured surfaces were etched with hot *o*-xylene in order to dissolve the dispersed PE phase present at the fracture surface. The etched surfaces were then investigated using SEM after sputter coating as above. The cavities left after dissolving the PE phase were used as a measure of the PE phase domain sizes.

2.5. Thermal characterisation

The thermal properties of the blends were investigated by differential scanning calorimetry (DSC) using a Mettler DSC3000 system equipped with a low-temperature cell. The samples, 10–15 mg, were sealed in 40 μl Al crucibles. The samples were heated from 25 to 245°C, cooled to 0°C, and then reheated to 245°C. Temperature scan rates were in all cases 10°C/min.

2.6. Dynamic mechanical properties

The dynamic mechanical properties were measured with a DMA 2980, instrument from TA Instruments Ltd. A single cantilever load cell was used. The frequency used was 1 Hz, and the amplitude was 3 μm. The temperature interval measured was from −100 to 100°C for the 70/30 blends and from −100 to 150°C for the 30/70 blends. The temperature scan rate was 3°C/min in all measurements.

3. Result and discussion

In a polymer blend, the morphology and the adhesion between the phases are determined by the interfacial interactions between the blend components. The material obtained by blending of PE and PA-6 has a very unstable morphology because of the large dissimilarity of the two polymers. PE is a pure hydrocarbon polymer with low polarity, while PA-6, because of its amide linkages, is quite polar and hydrogen bonding. The interfacial energy should consequently be high and the state of mixing should be very sensitive to changes in shearing rates at temperatures where both polymers are in the molten state.

To decrease the interfacial free energy of a blend, a surface-active component, for example, a block or graft copolymer, may be added as a compatibiliser. The compatibiliser collects at the interface and stabilises the blend morphology. However, in order to significantly improve the adhesion between the two phases, the different chain segments of the copolymer should be miscible or partly miscible with the blend components. To compatibilise polyamide blends, it is possible to use copolymers containing PEO. It has been shown that PEO is partly miscible with PA-6 due to hydrogen bonding interactions between the ether oxygen in PEO and the amide N–H groups in PA-6 [13–15]. The partial miscibility of PEO and PA-6 has been studied by Jannasch et al. [13] and Hu and co-workers [14–16].

In the present study, we have investigated the morphology and thermal properties of PE and PA-6 blends compatibilised with PE-PEO. Two different PE-PEO graft copolymers were utilised as compatibilisers. The copolymers had grafts with molecular weights of 750 and 2000 g/mol, respectively, and with similar total PEO contents (40 and 37%, respectively).

3.1. Blend morphology

Blends were prepared by melt mixing at 240°C of LDPE and PA-6 in the presence of various amounts of PE-PEO graft copolymer. After mixing, the blends were compression moulded into thin sheets, from which test specimens were cut. The specimens were then cryo-fractured at liquid N_2 temperature, and the effect of PE-PEO graft copolymers on the blend morphology was investigated. SEM micrographs of freeze fractured surfaces of the 70:30 blends are shown in Fig. 1a–c. Fig. 1a shows a binary blend, i.e. a blend prepared without any compatibiliser, and the micrograph clearly shows that the PA-6 phase is dispersed in the PE matrix as both spherical and fibre-shaped domains. It is also obvious from the micrograph that, as expected, the adhesion between the PE matrix and the PA-6 phase domains is poor. In a ternary, compatibilised, blend containing 4% PE-PEO2000 (Fig. 1b), the adhesion of the PA-6 domains to the PE matrix appears to be much stronger. The individual PA-6 domains, visible at the surface, are covered

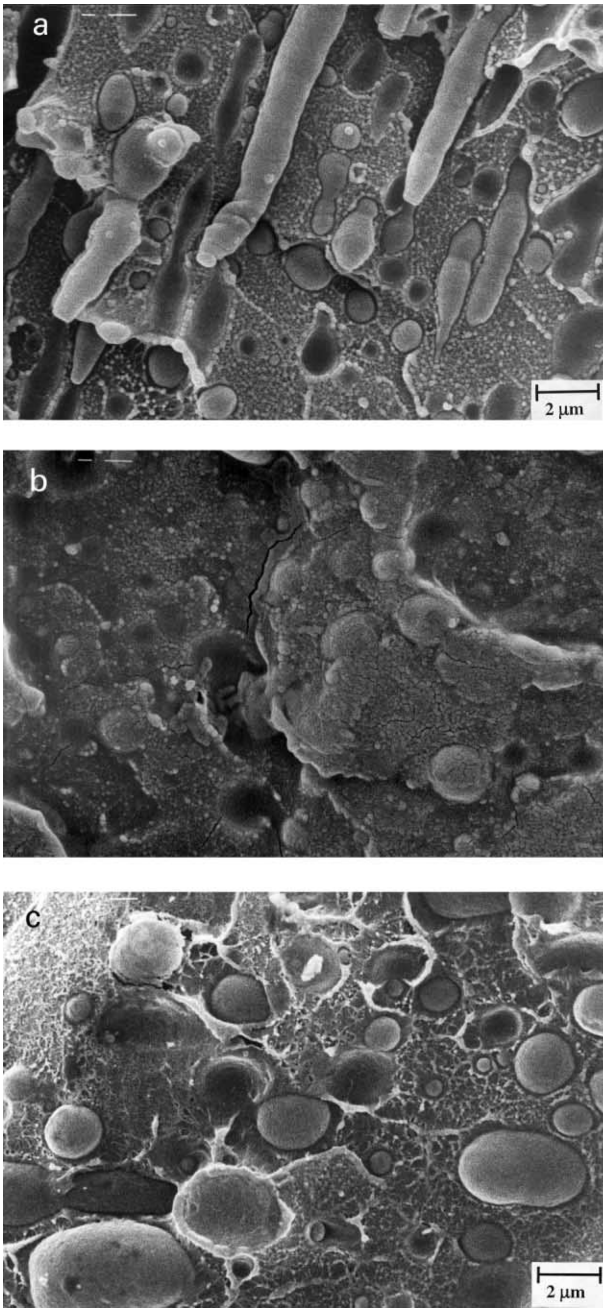

Fig. 1. SEM micrographs of freeze fractured PE/PA blend samples, compression moulded at 170°C: (a) PE/PA 70:30, (b) PE/PA 70:30 containing 4% PE-PEO2000, and (c) PE/PA 70:30 containing 4% PE-PEO750.

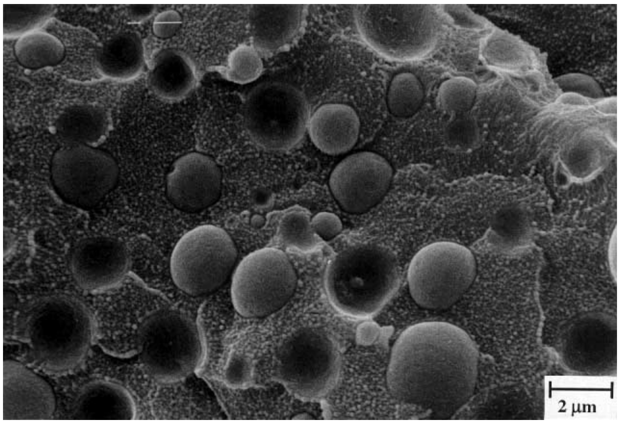

Fig. 2. SEM micrographs of freeze fractured PE/PA 70:30 blend, compression moulded at 240°C.

by matrix material. In the blend containing 4% PE-PEO750 (Fig. 1c), the adhesion between the phases seems to be as poor as in the binary blend.

The poor stability of the morphology in the binary blend is demonstrated by the micrograph in Fig. 2. After compression moulding at 240°C, the PA-6 phase has formed large, spherical domains with a weak adhesion to the matrix. At the moulding temperature, both phases were in the molten state. As a contrast, the morphology of the blend compatibilised with 4% PE-PEO2000 was unaffected by moulding at 240°C.

In order to determine the phase inversion composition, blends of PE/PA-6 in ratios of 60:40, 50:50 and 40:60 were prepared. The morphology of freeze fractured samples was studied by SEM, and as seen in Fig. 3, both the 60:40 and 50:50 blends displayed a continuous PE phase and PA-6 dispersed phase (Fig. 3a and b). A similar structure was also found in the compatibilised blends, but the adhesion between the phases was better. However, the blend with a PE/PA-6 ratio of 40:60 appeared to consist of two continuous phases (Fig. 3c). A similar blend containing 4% PE-PEO2000 is shown in Fig. 3d. In this blend ,the fracture surface was much smoother compared to that of the binary blend, and the phase borders were difficult to observe in the micrograph, indicating an increased adhesion between the PE and PA-6 phases. To get more information on the morphology of the 40:60 blends, the fracture surfaces were etched with hot *o*-xylene in order to dissolve the PE phase. SEM micrographs of the etched surfaces are shown in Fig. 4a and b. The PA-phase seems to have similar structures in both the binary and the compatibilised blends.

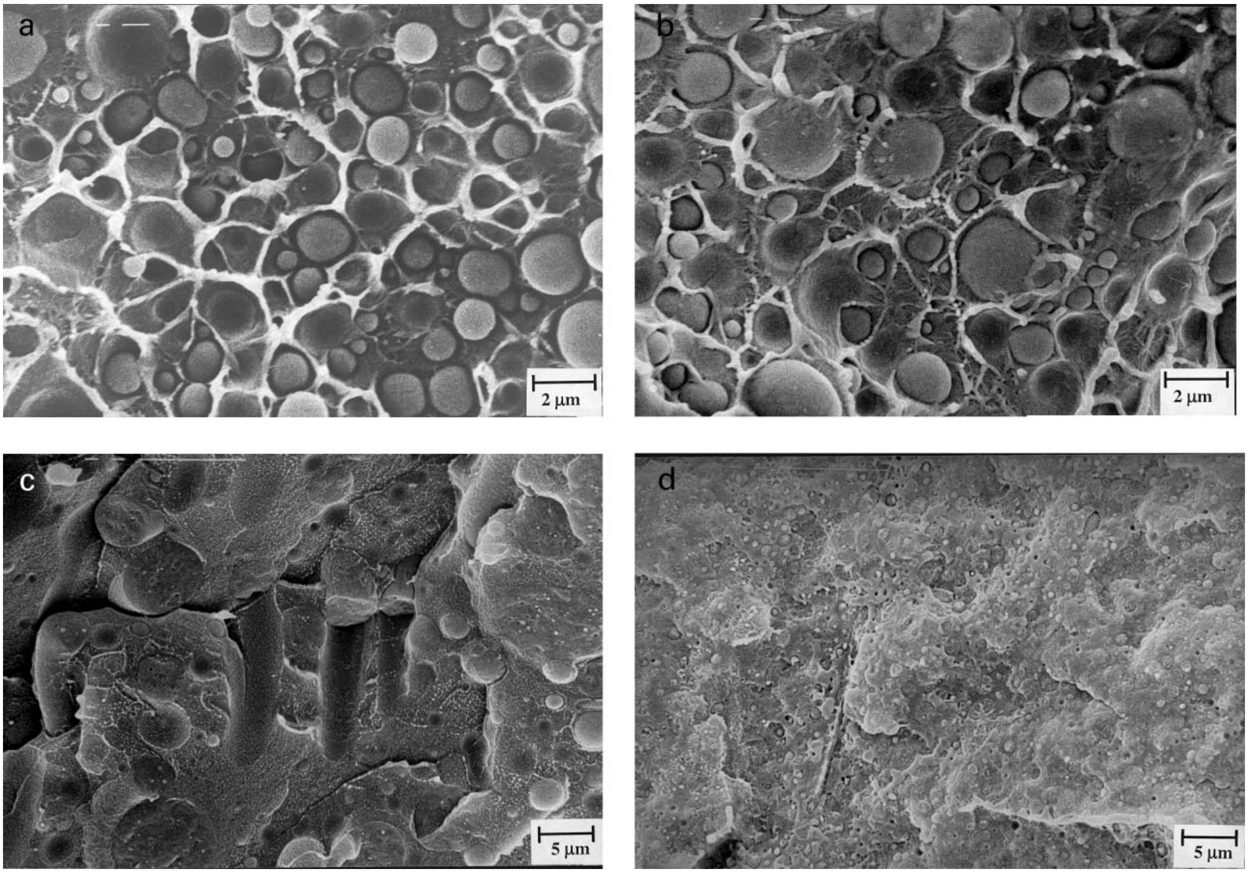

Fig. 3. SEM micrographs of freeze fractured PE/PA blend samples: (a) PE/PA 60:40, b) PE/PA 50:50, (c) PE/PA 40:60, and (d) PE/PA 40:60 containing 4% PE-PEO2000.

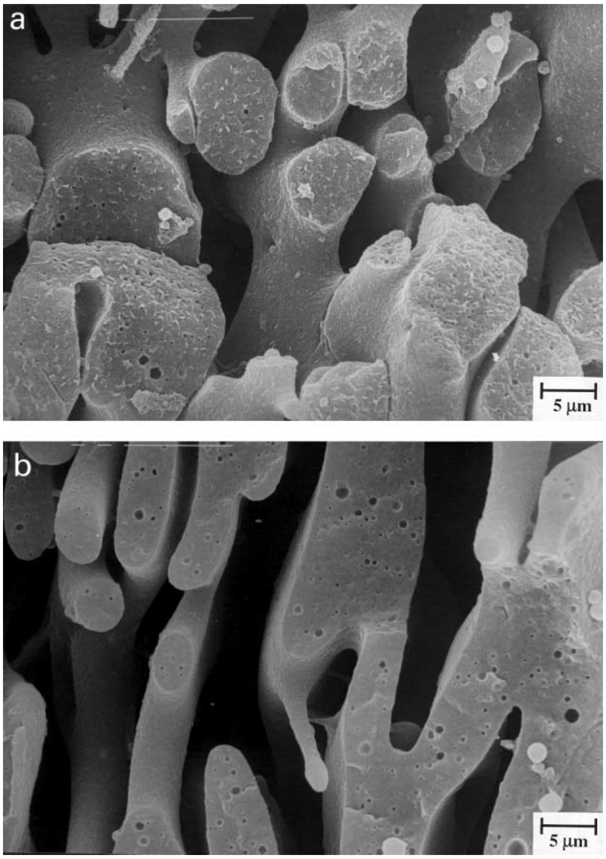

Fig. 4. SEM micrographs of freeze fractured PE/PA blend samples etched with hot *o*-xylene: (a) PE/PA 40:60, and (b) PE/PA 40:60 containing 4% PE-PEO2000.

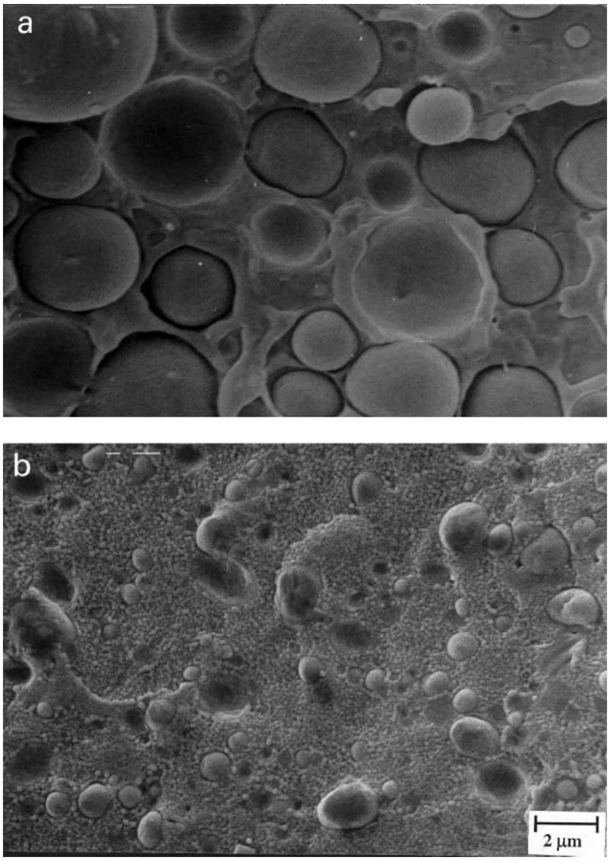

Fig. 5. SEM micrographs of freeze fractured PE/PA blend samples: (a) PE/PA 30:70, and (b) PE/PA 30:70 containing 4% PE-PEO2000.

Changing the composition of the PE/PA-6 to 30:70 changed the morphology of the blend. At this composition, PA-6 appeared as the continuous phase with a dispersed PE phase. In order to determine the effects of the addition of graft copolymer, blends containing increasing amounts of PE-PEO2000 were prepared. Micrographs of freeze fractured 30:70 blends are shown in Fig. 5. The binary blend shows a very weak adhesion between the phases, as noted for the binary 70:30 blend. The PE domains have a broad size distribution in the sample. In the blend compatibilised with PE-PEO2000 (Fig. 5b), the adhesion between the phases appears to be much stronger and the PE domains have significantly reduced sizes. It can thus be concluded that the PE-PEO2000 copolymer has similar compatibilising effects in both the 70:30 and 30:70 compositions.

3.2. Evaluation of the dispersed phase size

To evaluate the PA-6 domain size in the blends, the PA-6 phase domains were recovered by dissolving the PE matrix in hot *o*-xylene. The isolated PA-6 phase was transferred to sample holders and the average size and size distribution was evaluated using SEM. This method also gave an opportunity to study the three-dimensional shape of the PA-6 phase domains, which was very difficult in the freeze fractured samples. In Fig. 6, a micrograph of recovered PA-6 domains from a binary 70:30 blend is shown. The diameters of the individual PA-6 domains were evaluated from the micrographs. For each sample, approximately 2000 domains were measured. To evaluate the dispersed PE domain sizes in the PE/PA-6 30:70 blends, freeze fractured surfaces were etched with hot *o*-xylene and studied by SEM. The sizes of the cavities remaining after

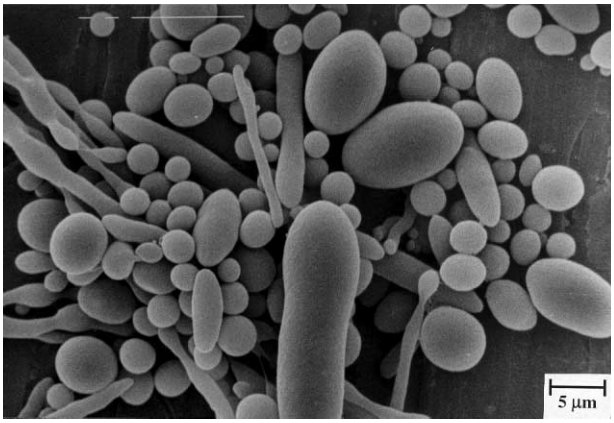

Fig. 6. SEM micrograph of isolated PA-domains from a PE/PA 70:30 blend.

dissolving the PE phase were evaluated from the micrographs and used as a measure of the dispersed phase size.

In Fig. 7a and b, the sizes of the dispersed phase domains are plotted as a function of the amount of PE-PEO2000 copolymer added to the PE/PA-6 blends. The weight average diameter, $\bar{D}_w$, decreased steeply when between 0 and 1% copolymer was added to the 70:30 blend, and at a level of 4%, the curve flattened out. This result indicates that the blend interfaces became saturated with the graft copolymer. For the 30:70 blends, the decrease in domain size was less steep at low concentrations of the graft copolymer. The characteristics of the curves and the saturation effect are common for compatibilised immiscible blends [5,17]. At low concentrations of the compatibiliser, the domain size of the dispersed phase is decreased due to a reduction of the interfacial tension. When the compatibiliser is added beyond the saturation concentration, the domain size does not decrease any further because the final domain size will now be determined by a balance of shear forces, which causes domain break-up, and interfacial tension, which opposes domain break-up and deformation [5,13].

To obtain more information on the size distribution of the dispersed phase, the cumulative distributions of the domain sizes in the PE/PA-6 blends were calculated. The results are given in Fig. 8a and b. The addition of PE-PEO2000 both reduced the domain sizes of the dispersed phase and narrowed the size distribution. The difference between the cumulative distributions for 4 and 6% PE-PEO2000 in the PE/PA-6 70:30 blends was small (Fig. 8a). This finding indicates that the saturation concentration of PE-PEO2000 presumably was on the order of 5% for the 70:30 blend. Excess copolymer will form a separate phase, which must be present either in the PE matrix or in the PA-6 phase. Due to the hydrogen bonding interactions between PEO and PA-6, it is likely that it will be present in the PA-6 phase. The most probable configuration of the copolymer is as aggregates with the MPEO chains phasing the PA-6 phase. Previous studies of PE-PEO2000 and LDPE blends

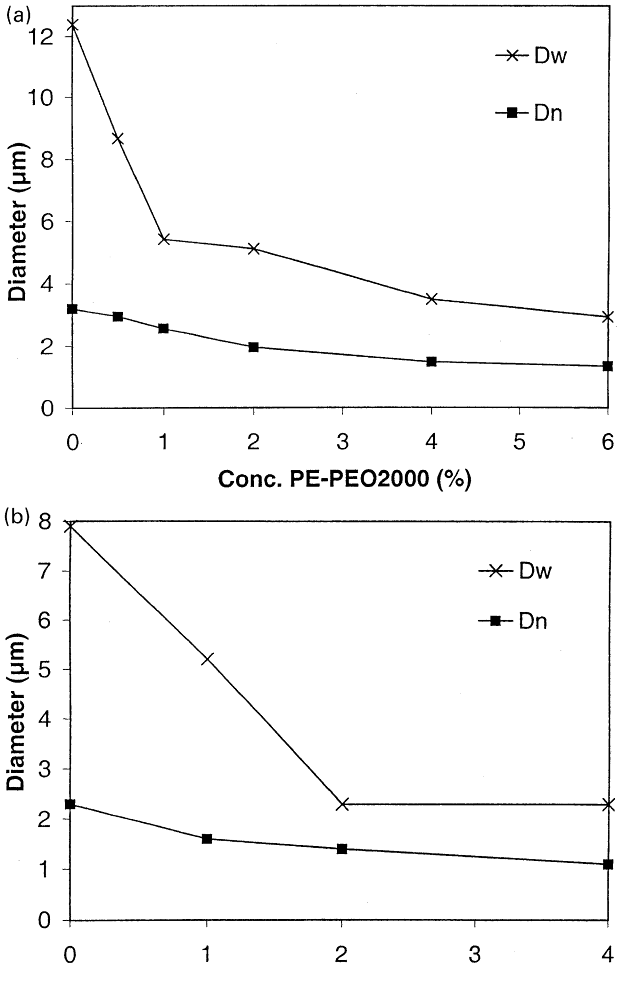

Fig. 7. Plots of dispersed phase size versus concentration of PE-PEO2000 in PE/PA blends: (a) 70:30 blend, and (b) 30:70 blend.

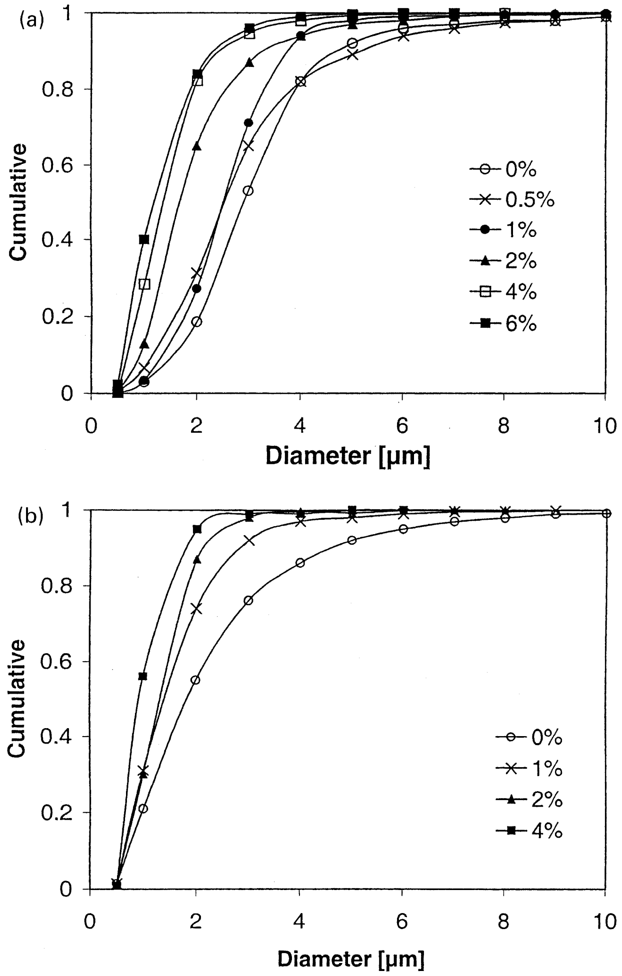

Fig. 8. Cumulative distributions of dispersed phase sizes in PE/PA blends containing various amounts of PE-PEO2000: (a) PE/PA 70:30, and (b) PE/PA 30:70.

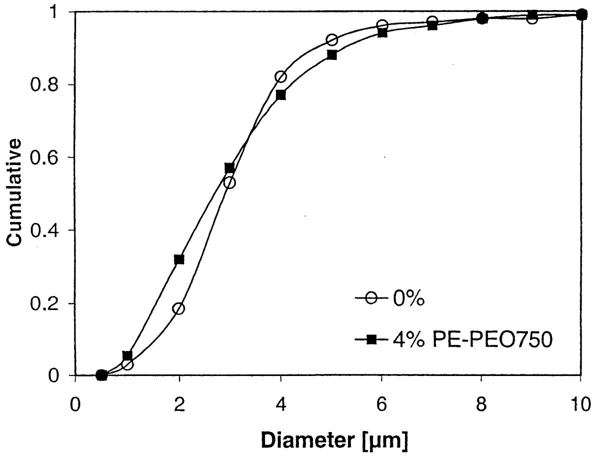

Fig. 9. Cumulative distributions of dispersed phase size in PE/PA 70:30 blends containing 0 and 4% PE-PEO750, respectively.

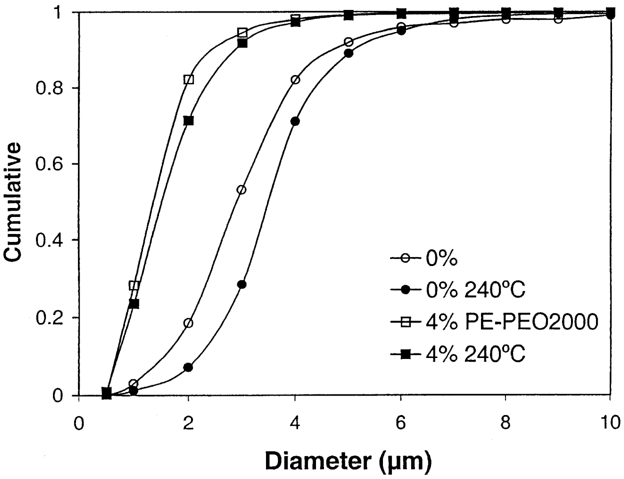

Fig. 10. Effect of compression moulding at 240°C on the dispersed PA phase. Cumulative distributions of the dispersed phase size in PE/PA 70:30 blends containing 0 and 4% PE-PEO2000, respectively.

have shown that the graft copolymer has a strong tendency to phase separate from LDPE [19].

Addition of 4% PE-PEO750 to the 70:30 blend did not affect the average size and the size distribution of the PA-6 domains, quite contrary to the addition of PE-PEO2000. This fact is obvious when comparing the cumulative distributions of the PA-6 phase from a binary PE/PA-6 blend and a compatibilised blend containing 4% PE-PEO750 (Fig. 9). The two distributions are quite similar, except that the blend containing PE-PEO750 has a slightly broader distribution. The two copolymers differ in the length of the grafts and the length of the PE segments between the grafts in the backbone, PE-PEO750 having the shorter grafts and the shorter segments of PE between the grafts. The grafts and backbone segments in PE-PEO750 are presumably too short to promote any significant interfacial activity in the blend, which is needed for the copolymer to be effective as a compatibiliser [13,18]. On the other hand, the longer side-chains and main chain segments present in the PE-PEO2000 graft copolymer should give a higher interfacial activity. In a previous article [19], we reported on the segregation of the graft copolymer to glass and air interfaces in binary blends of PE-PEO graft copolymers and LDPE. In that study, we observed that PE-PEO2000 was more surface selective than PE-PEO750, and enriched to a higher extent at the more polar glass/LDPE interface, as confirmed by XPS and water contact angles.

The effects of compression moulding on the PA-6 phase in 70:30 blends can be seen in Fig. 10, where the distributions for virgin blends and compression moulded blends are presented. At the moulding temperature (240°C) both phases were in the molten state. The moulding of the binary blend resulted in a shift of the distribution to larger PA-6 domains, which should be a result of coalescence of the PA-6 phase domains. The blend compatibilised with 4% PE-PEO2000 was clearly more stabilised against coalescence. As evident from Fig. 10 the difference between the distributions of virgin and moulded blends was small; only a minor increase in PA-6 domain size was observed.

In all PE/PA-6 blends with a weight ratio of 70:30 in addition to spheres and ellipsoids of PA-6, a continuous network of thin PA fibres having diameters of 0.4–2 μm was present, as seen in Fig. 11. It can be assumed that these fibrils constitute a transition between a fibrous and a spherical domain shape. This is indicated by string-of-beads appearance of the fibres. The continuous network structure was not possible to observe in the freeze-fractured surfaces and became visible only after dissolving the PE matrix. It is possible that the mixing time was too short or the mixing rate too low to reach a proper mixing. A higher mixing rate would presumably have given higher shear rates resulting in a break-up of the continuous fibre structure.

3.3. Thermal characterisation

Melting and crystallisation temperatures for the blends

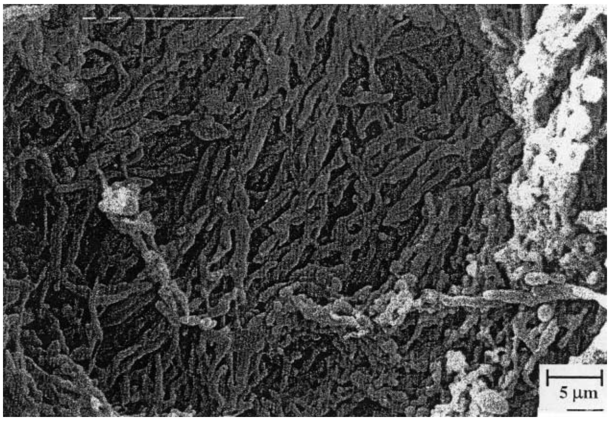

Fig. 11. Continuous network of isolated PA fibres from PE/PA 70:30.

Table 3
DSC melting enthalpies from the 1st and 2nd heating scan and crystallisation enthalpies for the PA-6 phase. All ΔH values were calculated on the PA-6 content in the sample

	T_m(PA) 1st (°C)	ΔH_m(PA) 1st (J/g)	T_m(PA) 2nd (°C)	ΔH_m(PA) 2nd (J/g)	T_c(PA) (°C)	ΔH_c(PA) (J/g)
PE/PA/PE-PEO2000						
PA-6	–	–	220	52	171	52
70/30	222	50	221	47	189	52
70/30/0.5	221	53	220	46	189	51
70/30/1	221	46	220	44	189	47
70/30/2	221	50	220	48	189	52
70/30/4	221	50	220	46	189	50
70/30/6	221	44	220	39	187	38
30/70	222	42	220	43	190	48
30/70/4	220	52	221	52	190	58
PE/PA/PE-PEO750						
70/30/4	220	46	220	44	190	51

and the corresponding crystallisation enthalpies were determined using DSC. The results from these measurements are presented in Tables 3 and 4. Pure PE and PA-6 show melting endotherms at 113 and 220°C, respectively, and crystallisation exotherms at 100 and 171°C. In the binary blends, as well as in the compatibilised PE/PA-6 blends, crystallisation of the PA-6 phase occurred at 189–190°C, i.e. 19°C higher than in pure PA-6. The PE/PA-6 70/30 blend containing 6% PE-PEO2000 was an exception having a T_c(PA) of 187°C. It appears as though the presence of the PE phase changes the nucleation rate, and allows crystallisation of the PA-6 phase at a higher temperature in the blends compared to pure PA-6. The melting temperature of the PA-6 phase was not affected by the blending and was registered at 220°C for the pure materials as well as for all the blends. The crystallisation and melting enthalpies for the PA-6 phase were approximately the same or slightly lower in the blends as compared to those of pure PA-6, also in this respect, the blend containing 6% PE-PEO2000 differed, showing lower crystallisation and melting enthalpies. The deviations noted for that blend as compared to the other ones indicate that at copolymer concentrations above the saturation concentration, which was approximately 5%, the excess graft copolymer would be present in the PA-6 phase rather than in the PE phase. As discussed above, interactions between PA-6 and PEO might favour this location of the copolymer. As for the PE phase in the blends, the crystallisation and melting behaviours was unchanged compared to that of pure PE.

3.4. DMA measurements

Dynamic mechanical analysis was performed on pure polymers and on blends, both binary and compatibilised 70:30 and 30:70 blends were investigated. The results from these measurements are shown in Fig. 12. The differences between the curves for binary blends and blends containing 4% PE-PEO2000 were very small at both PE/PA-6 ratios. It can be noted that the matrix material dominates the DMA curves, i.e. curves for the 70:30 blends strongly resemble that of pure PE, and curves for the 30:70 blends that of PA-6.

Table 4
DSC: melting enthalpies from the 1st and 2nd heating scan and crystallisation enthalpies for the PE phase. All ΔH values were calculated on the PE content in the sample

	T_m(PE) 1st (°C)	ΔH_m(PE) 1st (J/g)	T_m(PE) 2nd (°C)	ΔH_m(PE) 2nd (J/g)	T_c(PE) (°C)	ΔH_c(PE) (J/g)
PE/PA/PE-PEO2000						
PE	–	–	113	88	100	109
70/30	116	87	114	91	100	105
70/30/0.5	116	85	113	88	101	105
70/30/1	116	88	113	92	101	101
70/30/2	116	87	113	92	101	107
70/30/4	116	83	113	86	99	103
70/30/6	116	91	113	95	100	115
30/70	114	65	112	66	101	85
30/70/4	113	73	112	70	100	102
PE/PA/PE-PEO750						
70/30/4	113	62	115	59	100	72

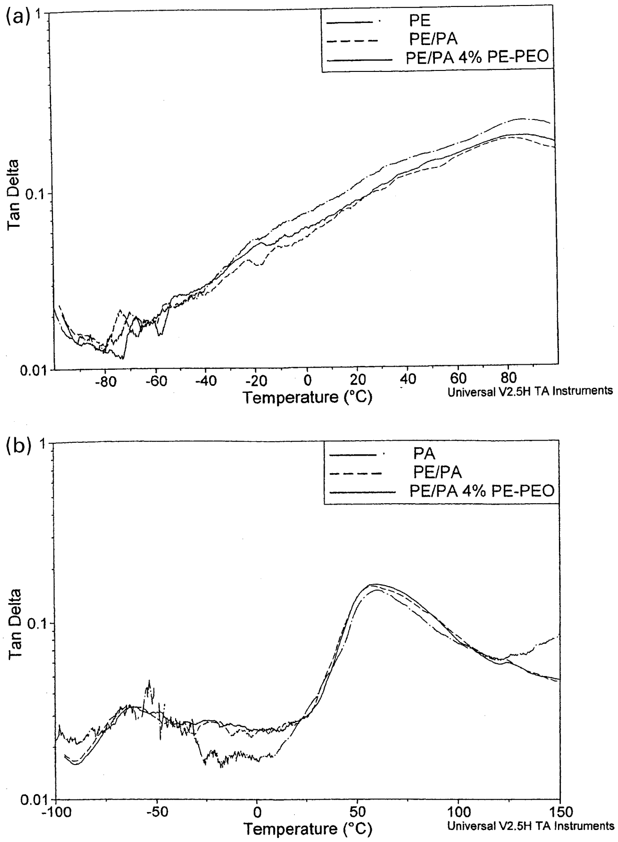

Fig. 12. DMA measurements: (a) pure PE and PE/PA 70:30 blends containing 0 and 4% PE-PEO2000, and (b) pure PA-6 and PE/PA 30:70 blends containing 0 and 4% PE-PEO2000.

4. Conclusions

The PE-PEO2000 graft copolymer was effective as compatibiliser for PE/PA-6 blends. The average size of the dispersed phase decreased significantly by the addition of the copolymer. The adhesion between the dispersed phase and the matrix was also significantly increased. At a PE/PA-6 ratio of 40:60, the polymers formed two continuous interpenetrating phases. The addition of 4% PE-PEO2000 to this blend improved the adhesion between the two phases significantly. The PE-PEO750 graft copolymer proved inefficient as compatibiliser for the PE/PA-6 70:30 blend. Addition of 4% PE-PEO750 to the blend had no measurable effect of the dispersed PA-6 domain size distribution.

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